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CERTIFICATE

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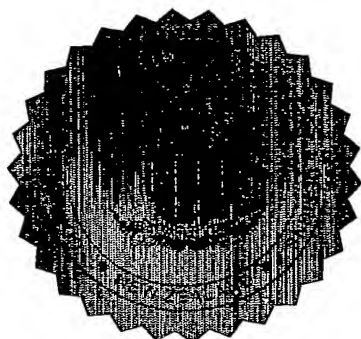
I hereby certify that annexed is a true copy of the Provisional Specification as filed on 22 July 2002 with an application for Letters Patent number 520369 made by The University of Waikato.

I further certify that pursuant to a claim under Section 24(1) of the Patents Act 1953, a direction was given that the application proceed in the name of TITANOX DEVELOPMENT LIMITED.

Dated 1 August 2003.

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James & Wells ref: 120030/31

PATENTS ACT 1953
PROVISIONAL SPECIFICATION

A SEPARATION PROCESS

We, The University of Waikato of Gate 5, Hillcrest Road (no number),
 Hamilton, New Zealand, a body existing by virtue of the University of
 Waikato Act 1963

do hereby declare this invention to be described in the following statement:

James & Wells Ref: 120030/31

A SEPARATION PROCESS

TECHNICAL FIELD

This invention relates to a separation process.

In particular, this invention relates to a method of separation of a component from a composite.

BACKGROUND ART

Titanium alloys have been widely used in making components for aircraft, medical implants and chemical processing machinery and structures.

Titanium alloys can also be used to replace steel in making automotive components, but this application has been severely limited by the high cost of titanium alloys.

This high cost is largely a result of the expensive batch processes that are used to recover titanium from its mineral concentrates, and the technical difficulties associated with melting and alloying titanium.

The conventional titanium production process the Kroll process which involves the reaction of TiO_2 and carbon, in the form of coke, under chlorine gas at temperatures of 800°C to form TiCl_4 and carbon monoxide.

The titanium chloride (TiCl_4) produced by this reaction exists as a liquid and has to be purified by distillation. The liquid is introduced into a furnace holding a magnesium melt at 680 to 750°C to facilitate the formation of magnesium chloride (MgCl_2) and pure titanium.

MgCl_2 is a gas, while titanium is a solid sponge. The sponge is purified by distillation or leaching using hydrochloric acid. The magnesium chloride can be recycled through an electrolysis process.

The titanium sponge that is formed by this process can be further processed to produce commercial purity titanium or titanium alloys by vacuum arc melting or other melting methods.

If titanium or titanium alloy powder is needed, the titanium or titanium alloy needs to be heated to a high temperature above 1650°C to produce titanium alloy melt and the alloy melt is atomised into liquid droplets which in turn solidifies as powders.

The limitations of this process include its complexity and the use of chlorine and magnesium. The process involves several high temperature steps where a high amount of energy is needed. This contributes to the high cost of titanium and titanium alloys. The use of chlorine makes the process environmentally unfriendly. Magnesium metal is expensive, so the use of magnesium in the process also contributes to the high cost of titanium. The result of this process is that the cost of titanium alloy powder is in the range of approximately US\$40 per kilogram.

United States Patent No. 6,264,719 (Zhang et al.) discloses both a titanium alloy based dispersion-strengthened composite and a method of manufacture of same. This patent discloses the use of dry high-energy intensive mechanical milling in the process of producing titanium based metal matrix composites (MMC).

High energy mechanical milling has the effect of providing the necessary number of small particles below the micrometer size range as well as enhancing the reactivity of different particles with one another.

While this patent has provided a method of producing titanium based MMCs at a reduced cost, it does not disclose a method for separating out unwanted components present within the MMC, or adjusting the level of certain components to more desirable concentrations. It would be an advantage over the present state of the art to have some way of removing unwanted components.

MMCs are composites of a tough conventional engineering alloy and a high strength second phase material, which may be an oxide, nitride, carbide or intermetallic. Oxide Dispersion Strengthened (ODS) alloys occur at one end of the spectrum of MMCs. These are composites of a tough engineering alloy and a fine dispersion of an oxide. Typically, in order to obtain the required dispersion, there must be no more than 10% volume fraction of the oxide second phase, which may have a size of 10's of nm.

All references, including any patents or patent applications cited in this specification are hereby incorporated by reference. No admission is made that any reference constitutes prior art. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinency of the cited documents. It will be clearly understood that, although a number of prior art publications are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning. For the purpose of this specification, and unless otherwise noted, the term 'comprise' shall have an inclusive meaning - i.e. that it will be taken to mean an inclusion of not only the listed components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process.

It is an object of the present invention to address the foregoing problems or at least to provide the public with a useful choice.

Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

DISCLOSURE OF INVENTION

According to one aspect of the present invention there is provided a method of separating a component from a metal based composite

characterised by including the step of

- a) increasing the size of the component to be separated.

The term 'component' in accordance with the present invention should be understood to mean any phase that makes up part of a metal based composite. While it should be appreciated that there is at least two components in a metal based composite, there is theoretically no limit to the number of components that make up a metal based composite.

The term 'metal-based composite' in accordance with the present invention should be understood to mean a metal matrix composite made up of at least two components where one is a metal.

In some embodiments the metal matrix composite could be termed a metal-metal composite, where the major metal component makes up greater than 50% of the final composite.

In other embodiments, the metal matrix composite could be termed a metal-ceramic composite where the major ceramic component makes up greater than 50% of the final composite and

In preferred embodiments, the metal based composite is a metal-ceramic composite where the combination of a metallic base and a reinforcing non-metallic constituent forms a single material. For ease of reference this term will now be used throughout the specification, however it should be appreciated that this term is not intended to be limiting.

The materials or phases that make up the metal-ceramic composite can include metallic phases, intermetallic phases, oxides, nitrides or carbides.

In preferred embodiments the components that make up the metal-ceramic composites are metallic phases, intermetallic phases and oxides, including Ti(Al,O) , $\text{Ti}_3\text{Al(O)}$ and TiAl(O) and Al_2O_3 ,

For ease of reference throughout the specification, Ti(Al,O) , $\text{Ti}_3\text{Al(O)}$ and TiAl(O) will now be collectively referred to as $\text{Ti}_x\text{Al}_y(\text{O})$. This term should not be seen as limiting.

In further preferred embodiments, the component to be separated from the metal-ceramic composite is Al_2O_3 . For ease of reference, the term Al_2O_3 will be used to refer to the component to be separated from the metal-ceramic composite, however, it should be appreciated that this should not be seen as being limiting and other components could be separated as preferred.

The combustion reaction used to produce $\text{Ti}_x\text{Al}_y/\text{Al}_2\text{O}_3$ composite from aluminium and titanium dioxide powders, as described in US Patent No. 6,264,719 results in the formation of Al_2O_3 particles and a titanium rich metallic or intermetallic phase.

The titanium metallic or intermetallic phase is either Ti(Al,O) which is a solid solution of Al and oxygen in titanium, or $\text{Ti}_3\text{Al(O)}$, or TiAl(O) which is a titanium aluminide intermetallic compound containing dissolved oxygen, or a mixture of these phases.

It is often a disadvantage of the method disclosed in US 6,264,719 that the volume fraction of the Al_2O_3 component in the $\text{Ti}_x\text{Al}_y/\text{Al}_2\text{O}_3$ composite is undesirably high, being greater than 45%. While Al_2O_3 is a desired component of a metal-ceramic composite; it is often desirable to reduce the volume fraction of Al_2O_3 to a low level of less than 15%.

By reducing the volume fraction of Al_2O_3 in the composite, mechanical properties of the metal-ceramic composites such as ductility and fracture toughness can be improved.

More importantly, by reducing the volume fraction of Al_2O_3 to less than 15%, the $\text{Ti}_x\text{Al}_y/\text{Al}_2\text{O}_3$ powder can be further reduced by using calcium to titanium alloy in titanium aluminide, thus providing an alternative route for producing these high value titanium base metallic materials.

In one preferred embodiment, the mean particle size of the Al_2O_3 is increased by heat treatment, which brings about coarsening of the Al_2O_3 particles.

The term 'particle' in accordance with the present invention should be understood to mean the individual embedded particles that make up a material and is a term known to someone skilled in the art. The shape of a particle within a solid is usually controlled by the presence of surrounding matrix and the application of heat to the material will allow the particles to coarsen, or grow in size.

By heating the $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ bulk composite to a temperature range of 1500-1650°C and holding at that temperature for a set period of time, ranging from 0.5-10 hours, the Al_2O_3 particles are significantly coarsened. The Al_2O_3 particle size increases to the range of approximately 15-40 μm .

It is an advantage of the present invention that the mean particle size of Al_2O_3 can be increased. By coarsening the Al_2O_3 particles in the composite, the material becomes more favourable for the later separation steps. This is contrary to conventional wisdom as the coarsening of embedded particles within a composite is usually undesirable, as coarsened particles can decrease the overall strength of the final product.

In order to facilitate the separation of Al_2O_3 from $\text{Ti}_x\text{Al}_y(\text{O})$, the composite with the

coarsened Al_2O_3 particles can then be crushed and milled by a milling means to produce a $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ powder. The majority of the powder particles (greater than 85% in volume) must be either Ti rich metallic particles or Al_2O_3 particles.

In preferred embodiments, this milling means is a high-energy mechanical mill.

In some embodiments, the milling of the composite is undertaken under an inert environment. This could include an inert atmosphere such as argon, or a vacuum.

In preferred embodiments, the inert atmosphere is argon.

The milling condition needs to be controlled in such a way that the composite microstructure is broken into Al_2O_3 and $\text{Ti}_x\text{Al}_y(\text{O})$ powder particles.

In preferred embodiments the milling time is limited in order to prevent the milling of Al_2O_3 beyond the preferred particle size.

The preferred particle size of Al_2O_3 is approximately the size of the Al_2O_3 particles produced after grain coarsening. It is a feature of each Al_2O_3 particle that after coarsening the structure of the particle is often single crystal. A single crystal material lacks or has no microstructure, including grain boundaries. It is a feature of single crystal that the lack of defects increases overall strength and single crystal Al_2O_3 is therefore hard and resists crushing. The metallic or intermetallic phase on the other hand is easily milled in comparison to the Al_2O_3 and the average particle size of each component is therefore significantly different after milling.

In some embodiments the separation of Al_2O_3 could be undertaken by means of sieving or the like after milling of the metal-ceramic composite, however, this is listed by way of example only and should not be seen to be limiting in any way as the Al_2O_3 Ti rich components could also be separated by using electrophoresis and electrostatic techniques or chemical leaching means.

In preferred embodiments the separation of Al_2O_3 from other components of the crushed $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ powder is by sedimentation of the components in a liquid.

To prepare the suspension for sedimentation, the crushed $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ powder is milled together with a mixture of water and surfactant to ensure good coating of each powder particle by the surfactant molecules, creating a slurry.

In preferred embodiments the liquid that the components are suspended in is water with a pH in the range of 4-10. However it should be appreciated that this is listed by way of example only and should not be seen to be limiting in any way.

In some embodiments the surfactant is either Sodium Dodecyl Sulphate (SDS) ($\text{C}_{12}\text{H}_{25}\text{SO}_4^- \text{Na}^+$) or Polyacrylic acid (PAA) ($[-\text{CH}_2\text{CH}(\text{CO}_2\text{H})-]_n$), although these are listed by way of example only and should not be seen to be limiting in any way.

In preferred embodiments, the surfactant is Sodium Dodecyl Sulphate (SDS) ($\text{C}_{12}\text{H}_{25}\text{SO}_4^- \text{Na}^+$).

In other preferred embodiments, the ratio of the surfactant to powder is typically 1:10 by weight.

The coating of the surfactant onto the powder particles provides a number of advantages. While the sizes of the particles rich in different components of the composite have been differentiated by the grain coarsening process and further milling processes, if they were suspended in a liquid, the sedimentation rate would be too high to differentiate the sedimentation times of the two types of powder particles. The addition of the surfactant assists suspension of the small particles in the liquid for a longer time and ensures the break up of agglomerates, and thus allows the larger, heavier Al_2O_3 rich particles to fall, as the sediment, well ahead of the smaller $\text{Ti}_x\text{Al}_y(\text{O})$ rich particles.

Once the milled composite is sufficiently coated, the slurry can then be mixed with a

quantity of a liquid to make a suspension with a preferred powder concentration.

In some embodiments, the powder concentration in the suspension is 5-20g/litre.

In preferred embodiments, the powder concentration in the suspension is 10-15g/litre.

It should be appreciated that the term liquid could include water or a water based solution, although these are listed by way of example and should not be seen to be limiting in any way.

In preferred embodiments, the liquid is water.

In further preferred embodiments the liquid has a pH in the range of 4-10.

The suspension can then be poured into a column of height ranging from 0.3 to 10m, and allowed to settle.

In preferred embodiments, the settling time ranges from 30 minutes to four hours.

After settling for a period of time, the suspension, which now mainly contains $Ti_xAl_y(O)$ rich powder, is taken out of the column and the solid powder is separated from the liquid by filtering, centrifuge separation, or other typical liquid/solid separation methods to produce a $Ti_xAl_y(O)$ rich powder. The sediment mainly contains Al_2O_3 rich powder.

It should be appreciated that while the sediment contains Al_2O_3 , it is only rich in this component. The milling process will produce components of differing mean particle size, however, there will still be some Al_2O_3 and other components that have either withstood the milling process, or have been crushed beyond the preferred limit.

The $Ti_xAl_y(O)$ rich powder produced from the first round of column sedimentation typically contains 20-40volume percent of Al_2O_3 . To further reduce the volume fraction of Al_2O_3 in the powder, the powder can be compacted, sintered and

coarsened to produce a $Ti_xAl_y(O)/Al_2O_3$ composite again and the crushing, wet milling, mixing with a liquid and column sedimentation cycle can be repeated.

It should be appreciated that this process cycle may be repeated multiple times to reduce the volume fraction of Al_2O_3 to a level typically in the range of 10-20%.

It should also be appreciated that the Al_2O_3 in the $Ti_xAl_y(O)$ rich powder may also be reduced by using other separating means such as electrophoresis or magnetic separation or the like, however these are listed by way of example only and should not be seen to be limiting.

In other embodiments of the present invention, $Ti_xAl_y(O)$ rich powder which has a volume fraction of Al_2O_3 less than 15% can be further mixed with calcium and heated to a temperature above $800^\circ C$ to facilitate the reaction between Al_2O_3 and calcium to consume the majority of Al_2O_3 , and between $Ti_xAl_y(O)$ and calcium to reduce the oxygen content in the $Ti_xAl_y(O)$ phase down to below 1.5 atomic percent. As a by-product of these reactions, the calcium oxide phase can be leached out using mild acids such as formic acid or the like, however, this is listed by way of example only and should not be seen to be limiting. In this way, a Ti-Al alloy or Ti_xAl_y compounds containing less than 1.5 atomic percent of dissolved oxygen can be produced.

It is an advantage to be able to produce a titanium based alloys or intermetallic compounds with an oxygen content below 1.5 atomic percent, as dissolved oxygen has a detrimental effect on the mechanical properties of these materials, and thus reduces their values accordingly.

Alternatively, the $Ti_xAl_y(O)$ rich powder containing less than 10 volume percent Al_2O_3 can be further mixed with some rare earth metals such as yttrium and cerium and sintered to allow the reaction of the rare earth metals with Al_2O_3 and $Ti_xAl_y(O)$ to form composites consisting of Ti_xAl_y with a low dissolved oxygen content (less than

1.5 atomic percent) and particles of rare earth oxides.

The addition of rare earth metals to the $Ti_xAl_y(O)$ rich powder allows to the production of high value specialised materials with certain desirable features depending on the rare earth metal added.

The term 'rare earth metal' is a term known to someone skilled in the art and refers to metals that appear in the lanthanide series of the period table of elements.

While the starting materials required to produce the final $Ti_xAl_y(O)$ rich composite, being TiO_2 powder, Al powder and other metal or metal oxide powders, should have purity levels of at least 98.5% to ensure the purity of the titanium alloys produced, it should be appreciated that in some cases, starting materials of lower level purity, such as the smelter slag from BHP New Zealand Steel Ltd can be used. Slag from BHP New Zealand Steel Limited typically contains 33 percent titanium oxide in moles.

It should be appreciated that a lower purity starting material may comprise the quality of the titanium based alloys or composites that are formed. When a less pure starting material is used, the processes may need to be modified to accommodate the lower purity starting materials.

It is an advantage of the present invention that $Ti_xAl_y(O)$ of high purity with limited and controllable amounts of Al_2O_3 present can be produced by a relatively low cost, environmentally friendly method of manufacture.

It is also an advantage of present invention that the separation technique does not utilise any environmentally restricted materials such as chlorine or the like.

It is also an advantage of the present invention that the production cost of high purity Ti_3Al is significantly reduced in comparison with existing production costs for Ti_3Al .

BRIEF DESCRIPTION OF DRAWINGS

Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawings in which:

Figure 1 The microstructure of $\text{Ti}(\text{Al},\text{O})/\text{Al}_2\text{O}_3$ composite produced by pressureless sintering of the Al/TiO_2 composite powder at different temperatures (a) 1550°C for 3 hours and (b) at 1650°C for 4 hours. The bright phase is $\text{Ti}(\text{Al},\text{O})$ and the dark phase is Al_2O_3 .

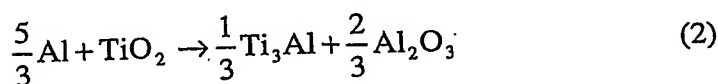
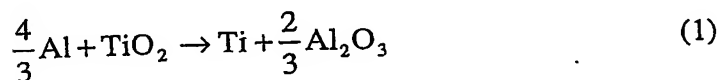
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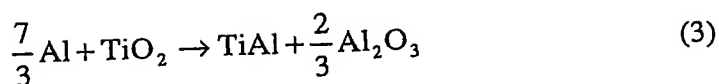
The steps detailed below utilise as a starting material, the Al/TiO_2 composite powder formed by the method of manufacture as disclosed in US Patent 6,264,719 and disclose the method of producing high purity Ti-Al alloy or Ti_xAl_y intermetallic compounds with Al_2O_3 in low and controlled concentration.

The starting materials used in the process, TiO_2 powder, Al powder and other metal or metal oxide powders, require a purity level of at least 98.5% to ensure the purity of the titanium alloys produced have the desired quality.

Step 1: Reaction sintering of the $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ bulk composite

The Al/TiO_2 composite powder produced using the method disclosed in US Patent 6,264,719 is pressed into a compact. The mole ratio between Al and TiO_2 can be controlled according to one of the following nominal expressions:





The compact is heated to a temperature sufficiently high to ignite the composition reaction between Al and TiO₂, forming Ti_xAl_y(O) including Ti(Al,O) and Al₂O₃. The heating and combustion reaction also causes sintering of the powder compact into a bulk Ti_xAl_y(O)/Al₂O₃ composite. The temperature required for ignition is 700°.

Alternatively, the Al/TiO₂ composite powder can be heated to a temperature above 700°C to allow the ignition of the combustion reaction between Al and TiO₂ forming a Ti_xAl_y(O)/Al₂O₃ composite powder.

The Ti_xAl_y(O)/Al₂O₃ composite powder can be pressed into a compact and sintered into a bulk Ti_xAl_y(O)/Al₂O₃ by holding the compact at 1550°C for 2 hours.

Step 2: Method of Coarsening of Al₂O₃ particle size

The bulk Ti_xAl_y(O)/Al₂O₃ bulk composite is heated to 1650°C and is held at this temperature for 4 hours to cause the Al₂O₃ particles in the Ti_xAl_y(O)/Al₂O₃ composite to be significantly coarsened, as shown in Figure 1. The size of the Al₂O₃ particles should be in the range of 15-40µm. The heating rate is 5°C/minute.

It should be appreciated that sometimes it might be advantageous to combine step 1 and step 2.

Step 3: Preparation of the composite for separation

Crush and mill the Ti_xAl_y(O)/Al₂O₃ composite with coarsened microstructure using a high-energy mechanical mill under argon or other inert atmosphere including vacuum. The milling time is 20 minutes. The milling condition needs to be controlled in such a way that the composite microstructure is broken into discrete Al₂O₃ and Ti_xAl_y(O) powder particles. The milling should not be too long and the reduction of Al₂O₃ particle size should be prevented. The size of the powder particles

is in the range of 0.5-100 μ m. The powder contains 40-60vol.% of Al_2O_3 depending on the Al/TiO_2 mole ratio in the starting powder.

Step 4: Addition of a surfactant to the powder & production of slurry

The $\text{Ti}_x\text{Al}_y(\text{O})/\text{Al}_2\text{O}_3$ powder produced from step 3 is further mixed with water and surfactant at a ratio of 1 gram of powder to 10 ml of water and 0.1 gram of surfactant in a low energy mechanical mill. The surfactant is sodium dodecyl sulphate. The milling time is 30 minutes, or sufficiently long to coat the powder particles surface with water and the surfactant molecules. At the end of this step, a slurry is produced.

Step 5: Separation of the slurry

The slurry from step 4 is mixed with a large quantity of water to make a suspension with a powder concentration of 10g/litre. The suspension is then poured into a column with a height of 5m. After a time of 1 hour, the suspension is taken out of the column. The powder in the suspension is separated from the liquid using a typical solid-liquid separation method such as filtering or centrifugal separation. The powder produced is called B1 powder. In the meantime, sediment at the bottom of the column is also taken out of the column and filtered. The powder produced from the sediment is called B2 powder. B1 powder, which accounts for typically 30-50% of the total powder, contains 20-40vol.% Al_2O_3 . B2 powder, which accounts for typically 50-70% of the total starting powder, contains 70-85vol.% Al_2O_3 . B1 powder has a particle size typically in the range of 0.5-10 μ m, while B2 powder has a particle size typically in the range of 5-100 μ m.

Step 6: Further separation of low Al_2O_3 powders

B1 powder is compacted by using mechanical presses and the material goes through step 1 to step 5 again to produce B3 and B4 powders. B3 powder is from the suspension and contains a lower volume fraction of Al_2O_3 than the B1 powder. The

volume fraction of Al_2O_3 particles in B3 powder is in the range of 15-25%. B4 is the by-product of the process. B1 to B4 powders are all valuable materials in their own right.

Steps 1 to 5 may be repeated more than once to produce B5 and B6, or B7 and B8 powders.

Step 6a: Extraction of titanium rich powder from suspension

The B1 or B3 powder produced from step 5 is mixed with surfactant and water to produce a suspension with a solid concentration of 10g/litre. The titanium rich powder is then extracted from the suspension by using either an electrophoresis process or a magnetic separation process.

Step 7: Addition of Ca to reduce oxygen content in titanium rich powder

Once the volume fraction of the Al_2O_3 in the $\text{Ti}_x\text{Al}_y(\text{O})$ rich powder is below 15%, the powder will be mixed with calcium (Ca), and heated to 800°C to facilitate the reaction between Al_2O_3 and Ca to eliminate Al_2O_3 and between $\text{Ti}_x\text{Al}_y(\text{O})$ and Ca to reduce the oxygen content in the $\text{Ti}_x\text{Al}_y(\text{O})$ phase down to below 1at%. As a by-product of the reaction, the CaO phase will be leached out using mild acids such as formic acid.

Step 7a: Addition of Y and Ce to titanium rich powder

Alternatively, once the volume fraction of the Al_2O_3 in the $\text{Ti}_x\text{Al}_y(\text{O})$ rich powder is below 10%, the powder will be mixed with rare earth metals such as yttrium (Y) and cerium (Ce) to react with Al_2O_3 and $\text{Ti}_x\text{Al}_y(\text{O})$ to form composites consisting of Ti_xAl_y with a low dissolved oxygen content (<1at%) and the particles of oxides of rare earth metals.

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof.

THE UNIVERSITY OF WAIKATO

by its Attorneys

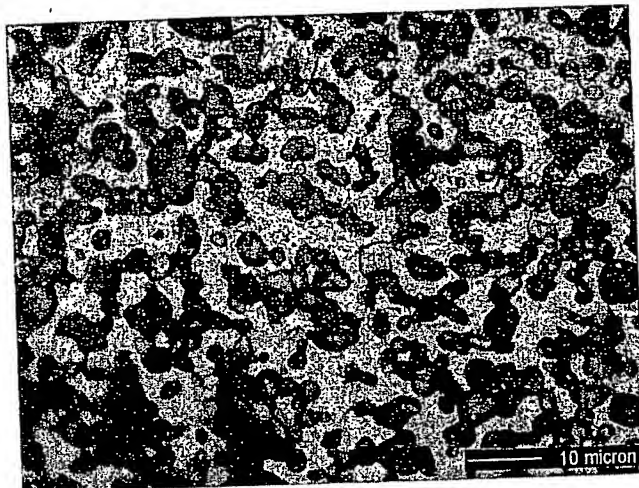
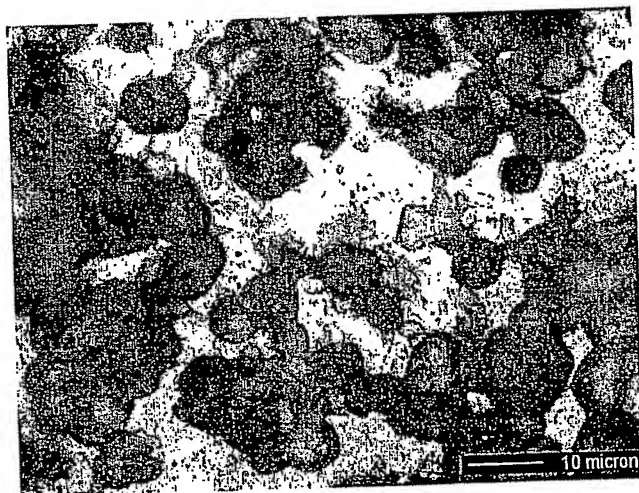


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